Crystal structure of dimethyl 1,1-diacetyl-8a-hydroxy-8-oxo-1,2,8,8a-tetrahydrocyclopenta[*a*]indene-2,3-dicarboxylate, C₂₀H₁₈O₈

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Abstract

C₂₀H₁₈O₈, orthorhombic, *Fdd2* (no. 43), *a* = 19.060(4) Å, *b* = 35.937(7) Å, *c* = 10.571(2) Å, *V* = 7240.6 Å³, *Z* = 16, *R*_{gt}(*F*) = 0.056, *wR*_{ref}(*F*²) = 0.139, *T* = 110 K.

Source of material

Dimethyl 1,1-diacetyl-8a-hydroxy-8-oxo-1,2,8,8a-tetrahydrocyclopenta[*a*]indene-2,3-dicarboxylate was synthesized in accordance with the published procedure [1]. Single crystals of the title compound were prepared in benzene at 322 K during six weeks using the branch tube method. The dark-yellow crystals were filtered off, washed with cold benzene (5 ml) and dried in vacuum over P₄O₁₀ (m.p. 453 K). Elemental analyses were consistent with the composition C₂₀H₁₈O₈ (found – C, 62.93 %; H, 5.08 %; O, 33.43 %; calc. – C, 62.18 %; H, 5.00 %; O, 33.16 %). Melting points were measured on an Electrothermal 9100 apparatus and are uncorrected. Elemental Analyses were performed using a Heraeus CHN-O-Rapid analyzer.

Discussion

Indene derivatives are important carbocycles in bio-organic chemistry and are present in many natural products and pharmaceuticals [1]. In recent years there has been increasing interest in the determination of crystal structures of biologically active compounds [2–8].

In the crystal structure of the title compound, the covalent bonds are found to be in normal range. Relevant bond lengths *d*(O8—C5) = 1.211(3) Å, *d*(O2—C15) = 1.221(3) Å, *d*(O3A—C4) = 1.437(3) Å, *d*(O7—C13) = 1.197(3) Å, *d*(O4—C14) = 1.338(3) Å, *d*(O4—C18) = 1.456(4) Å, *d*(O1—C16) = 1.211(3) Å, *d*(C1—C12) = 1.340(3) Å, *d*(C1—C13) = 1.488(3) Å, *d*(C5—C6) = 1.487(3) Å, *d*(O6—C13) = 1.349(3) Å and bond angles ∠C14—O4—C18 = 114.0(2)°, ∠C15—C3—C16 = 111.12(17)°, ∠O8—C5—C4 = 127.5(2)°, ∠C6—C5—C4 = 105.06(18)°, ∠O7—C13—O6 = 124.4(2) are similar to those in previous reports [2–8]. The methyl groups at O4 and O6 are approximately in the O4—C14—O5 (∠C18—O4—C14—O5 = 2.5(4)°) and O6—C13—O7 (∠C17—O6—C13—O7 = –6.2(4)°) planes, respectively, which agrees well with a participation of O4 and O6 in the electron donation to the esteric carbonyl groups. The torsion angle ∠C12—C1—C13—O7 = –12.0(4)° shows clearly that the carbonyl group C13=O7 and the double bond C1=C12 share approximately a common plane with *S-cis* conformation probably as a result of the π-system conjugation. Based on torsion angles C14 atom is in *trans* position relative to O3A. No π–π-ring stacking is observed within the crystals. This probably is a result of the presence of bulky groups in the crystal network which prevent stacking of the aromatic rings. Intermolecular hydrogen bonds between O2 and H1O(–O3A) atoms (*d*(O2...H1O) = 1.98 Å and ∠O2–H1O–O3A = 171.9°) are observed within the crystals, probably caused by the presence of electron-poor (H1O(–O3A)) and electron-rich (O2) atoms in suitable positions of the crystal network. These intermolecular interactions appear to be plausible factors in the stabilization of the crystal packing.

Table 1. Data collection and handling.

Crystal:	dark-yellow prism, size 0.23 × 0.25 × 0.40 mm
Wavelength:	Mo <i>K</i> _α radiation (0.71073 Å)
<i>μ</i> :	1.11 cm ^{–1}
Diffractometer, scan mode:	Bruker SMART 1000 CCD, <i>φ</i> / <i>ω</i>
2 θ _{max} :	58°
<i>N</i> (<i>hkl</i>) _{measured} , <i>N</i> (<i>hkl</i>) _{unique} :	19878, 4783
Criterion for <i>I</i> _{obs} , <i>N</i> (<i>hkl</i>) _{gt} :	<i>I</i> _{obs} > 2 σ (<i>I</i> _{obs}), 3820
<i>N</i> (<i>param</i>) _{refined} :	325
Program:	SHELXTL [9]

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Table 2. Atomic coordinates and displacement parameters (in Å²).

Atom	Site	<i>x</i>	<i>y</i>	<i>z</i>	<i>U</i> _{iso}
H(1)	16 <i>b</i>	0.211(2)	−0.0069(8)	0.606(3)	0.031(7)
H(2)	16 <i>b</i>	0.555(2)	0.031(1)	0.767(4)	0.07(1)
H(3)	16 <i>b</i>	0.259(2)	0.042(1)	0.453(4)	0.07(1)
H(4)	16 <i>b</i>	0.436(2)	0.0791(8)	1.123(3)	0.028(7)
H(5)	16 <i>b</i>	0.161(2)	0.0153(9)	0.685(3)	0.041(9)
H(7)	16 <i>b</i>	0.328(2)	0.0524(9)	0.500(3)	0.034(8)
H(8)	16 <i>b</i>	0.130(2)	0.119(1)	1.192(4)	0.06(1)
H(9)	16 <i>b</i>	0.179(2)	0.088(1)	1.258(4)	0.05(1)
H(10)	16 <i>b</i>	0.277(2)	0.083(1)	0.430(4)	0.06(1)

Table 2. Continued.

Atom	Site	<i>x</i>	<i>y</i>	<i>z</i>	<i>U</i> _{iso}
H(11)	16 <i>b</i>	0.610(2)	0.0363(8)	0.964(3)	0.039(8)
H(12)	16 <i>b</i>	0.016(2)	0.056(1)	0.879(4)	0.045(9)
H(13)	16 <i>b</i>	0.214(2)	0.1025(9)	0.828(3)	0.038(8)
H(14)	16 <i>b</i>	0.550(2)	0.064(1)	1.142(3)	0.041(8)
H(15)	16 <i>b</i>	0.192(2)	−0.0236(8)	0.737(3)	0.033(7)
H(17)	16 <i>b</i>	0.204(2)	0.131(1)	1.257(4)	0.05(1)
H(40)	16 <i>b</i>	0.051(3)	0.019(2)	0.898(5)	0.08(2)
H(19)	16 <i>b</i>	0.053(2)	0.048(1)	1.004(5)	0.06(1)
H(10)	16 <i>b</i>	0.384(2)	0.1234(8)	0.738(3)	0.020(6)

Table 3. Atomic coordinates and displacement parameters (in Å²).

Atom	Site	<i>x</i>	<i>y</i>	<i>z</i>	<i>U</i> ₁₁	<i>U</i> ₂₂	<i>U</i> ₃₃	<i>U</i> ₁₂	<i>U</i> ₁₃	<i>U</i> ₂₃
O(8)	16 <i>b</i>	0.42971(9)	0.03878(5)	0.6174(2)	0.0238(8)	0.0382(9)	0.0248(8)	0.0008(7)	0.0017(7)	−0.0057(7)
O(2)	16 <i>b</i>	0.19163(9)	0.09517(5)	0.6030(2)	0.0286(8)	0.0290(8)	0.0287(9)	0.0054(6)	−0.0074(7)	−0.0010(7)
O(3A)	16 <i>b</i>	0.35492(9)	0.11028(4)	0.6970(2)	0.0272(8)	0.0233(7)	0.0215(8)	−0.0053(6)	−0.0039(7)	0.0040(6)
O(7)	16 <i>b</i>	0.31178(9)	0.09248(5)	1.1699(2)	0.037(1)	0.0347(9)	0.0209(8)	−0.0004(7)	−0.0004(7)	−0.0015(7)
O(4)	16 <i>b</i>	0.11024(9)	0.06720(5)	0.8654(2)	0.0188(8)	0.045(1)	0.0344(9)	0.0016(7)	0.0024(7)	0.0043(8)
O(1)	16 <i>b</i>	0.31482(8)	0.00082(5)	0.7903(2)	0.0267(8)	0.0254(7)	0.0294(8)	0.0042(7)	−0.0028(7)	0.0048(6)
C(3)	16 <i>b</i>	0.2725(1)	0.06129(6)	0.7327(2)	0.0172(9)	0.0206(9)	0.018(1)	0.0000(7)	0.0006(8)	−0.0010(8)
C(16)	16 <i>b</i>	0.2675(1)	0.01825(6)	0.7419(2)	0.023(1)	0.0207(9)	0.020(1)	0.0014(8)	0.0048(8)	−0.0002(8)
O(5)	16 <i>b</i>	0.18359(9)	0.02912(5)	0.9698(2)	0.0300(9)	0.0345(9)	0.037(1)	0.0001(7)	0.0058(8)	0.0106(8)
C(15)	16 <i>b</i>	0.2437(1)	0.07539(5)	0.6052(2)	0.023(1)	0.0185(9)	0.023(1)	−0.0035(8)	−0.0046(9)	0.0025(8)
C(11)	16 <i>b</i>	0.4261(1)	0.06893(6)	0.9372(2)	0.022(1)	0.0216(9)	0.021(1)	−0.0021(8)	−0.0019(8)	0.0008(8)
C(4)	16 <i>b</i>	0.3491(1)	0.07439(6)	0.7559(2)	0.019(1)	0.0209(9)	0.019(1)	−0.0015(8)	−0.0018(8)	0.0008(7)
O(6)	16 <i>b</i>	0.2062(1)	0.10612(6)	1.0859(2)	0.037(1)	0.051(1)	0.0248(9)	0.0162(8)	0.0040(8)	−0.0034(8)
C(19)	16 <i>b</i>	0.2033(1)	−0.00004(6)	0.6879(3)	0.026(1)	0.022(1)	0.031(1)	−0.0018(9)	0.000(1)	−0.0003(9)
C(2)	16 <i>b</i>	0.2326(1)	0.07972(6)	0.8487(2)	0.022(1)	0.0212(9)	0.019(1)	0.0029(8)	0.0010(8)	−0.0021(8)
C(1)	16 <i>b</i>	0.2902(1)	0.08410(6)	0.9477(2)	0.026(1)	0.0186(9)	0.018(1)	0.0015(8)	−0.0001(8)	−0.0006(8)
C(8)	16 <i>b</i>	0.5647(1)	0.04546(7)	0.9563(3)	0.022(1)	0.031(1)	0.040(1)	0.0009(9)	−0.007(1)	0.000(1)
C(5)	16 <i>b</i>	0.4157(1)	0.05224(6)	0.7193(2)	0.024(1)	0.023(1)	0.024(1)	−0.0017(8)	0.0019(9)	−0.0001(8)
C(6)	16 <i>b</i>	0.4618(1)	0.05417(6)	0.8325(2)	0.022(1)	0.024(1)	0.023(1)	−0.0003(8)	−0.0021(8)	−0.0014(8)
C(13)	16 <i>b</i>	0.2732(1)	0.09410(6)	1.0807(2)	0.032(1)	0.0217(9)	0.022(1)	0.0017(8)	0.0039(9)	0.0000(8)
C(20)	16 <i>b</i>	0.2802(1)	0.06310(7)	0.4877(2)	0.033(1)	0.031(1)	0.019(1)	0.001(1)	−0.002(1)	0.000(1)
C(10)	16 <i>b</i>	0.4602(1)	0.07141(6)	1.0548(2)	0.031(1)	0.026(1)	0.022(1)	−0.0014(9)	−0.0047(9)	−0.0006(9)
C(12)	16 <i>b</i>	0.3540(1)	0.07746(6)	0.8993(2)	0.024(1)	0.0200(9)	0.020(1)	−0.0001(8)	−0.0035(8)	0.0001(8)
C(7)	16 <i>b</i>	0.5315(1)	0.04239(6)	0.8404(3)	0.023(1)	0.026(1)	0.034(1)	0.0004(8)	−0.001(1)	−0.0018(9)
C(14)	16 <i>b</i>	0.1737(1)	0.05553(7)	0.9023(2)	0.024(1)	0.031(1)	0.024(1)	0.0008(9)	0.0045(9)	−0.0025(9)
C(9)	16 <i>b</i>	0.5294(1)	0.05986(7)	1.0619(3)	0.032(1)	0.032(1)	0.030(1)	−0.002(1)	−0.011(1)	0.004(1)
C(18)	16 <i>b</i>	0.0524(2)	0.0459(1)	0.9187(5)	0.020(1)	0.087(3)	0.063(2)	−0.006(2)	0.005(1)	0.023(2)
C(17)	16 <i>b</i>	0.1789(2)	0.1139(1)	1.2105(3)	0.047(2)	0.063(2)	0.031(1)	0.018(2)	0.011(1)	−0.005(1)

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